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Small Angle X-Ray Scattering Study of the Structure and Ordering of Micelles in a Triblock Copolymer

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Beamline(s):X27C

Introduction: Block copolymers are known to form micelles in selective solvents, with the solvent-insoluble polymer block forming the micelle core and the solvent-soluble block the corona. Understanding of the kinetics of micelle formation and of micellar ordering is of fundamental and practical interest [1,2].

Methods and Materials: The sample of polystyrene(PS)-block-poly(ethylene-co-butylene)(PEB)-block-polystyrene triblock copolymer (Shell Chemicals, Kraton G-1650) with a molecular weight M_w of 100,000 was used in this study. Solutions in mineral oil and dioxane were prepared at different concentrations. Mineral oil is a good solvent for the middle PEB block, while dioxane is a good solvent for the outer PS block. Time-resolved small angle x-ray scattering (SAXS) measurements were performed at Beamline X27C of the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. SAXS measurements were made following a rapid temperature jump, or a slow temperature cooling ramp.

Results: Our results showed that in mineral oil the scattering function can be fit by the Percus-Yevick hard-sphere model. The core radius, the hard sphere interaction radius and the volume fraction of the hard sphere increase and then saturate as the temperature decreases. In the case of mineral oil solution, we observed that micelles undergo a transition from a disordered fluid state to a cubic arrangement of spherical micelles. Temperature jumps of different quench depth reveal a two-stage transformation kinetics, with the early stage development characterized by the growth of the primary peak and late stage by the emergence and rapid growth of higher order Bragg peaks. The duration of the first stage and the height of the peak reflect the influence of the quench depth on the transformation kinetics. From the cooling ramp we were able to estimate the transition temperature.

Conclusions: Kinetics of the transition from disordered micelles to cubic ordered micellar state was measured and the results were interpreted in terms of nucleation and growth models.

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